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## (54) SILICON SINGLE CRYSTAL WAFER AND ITS PRODUCTION

(57)Abstract:

**PROBLEM TO BE SOLVED:** To produce the silicon single crystal wafer in which latent nuclei of OSF (oxidation induced stacking faults) or ring-shaped OSF, that are to be caused at the time of subjecting the wafer to thermal oxidation treatment, are present in an extremely low density, however, any FPD(flow pattern defect), COP(crystal originated particle), L/D(large dislocation), LSTD (laser scattering tomography defect) or defect detected by Cu decoration, is not present throughout the whole surface of the wafer, under stable production conditions by using a CZ (Czochralski) method.

**SOLUTION:** In the production of a single crystal used as this wafer by using a CZ method, at the time of growing the single crystal, the single crystal is pulled up while controlling the furnace inside temp. so that  $\Delta G$  is 0 or a negative value (wherein:  $G$  ( $^{\circ}\text{C}/\text{cm}$ ) is the temp. gradient ((the amount of change in temp.)/ (length in the direction of the crystal axis)) in the vicinity of a solid-liquid interface in the crystal, within the temp. range of from the melting point to  $1,400^{\circ}\text{C}$ ;  $G_c$  ( $^{\circ}\text{C}/\text{cm}$ ) is the temp. gradient in the central part of the crystal;  $G_e$  ( $^{\circ}\text{C}/\text{cm}$ ) is the temp. gradient in the peripheral part of the crystal; and  $\Delta G$  is the difference between  $G_e$  and  $G_c$ , i.e.,  $\Delta G = (G_e - G_c)$ , and also controlling the single crystal pulling-up rate so as to fall within the range of from a pulling-up rate corresponding to the minimum value on the inner border line of the OSF region to a pulling-up rate corresponding to the minimum value on the outer border line of the OSF region, when an OSF region in the form of an inverted M-shaped belt is formed in a defect distribution diagram showing crystal defect distribution plotted with the crystal diameter and the crystal pulling-up rate as the abscissa and the ordinate respectively.

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**CLAIMS**

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[Claim(s)]

- [Claim 1] The silicon single crystal wafer characterized by the defect which it is the silicon single crystal wafer raised by the Czochralski method, the nucleus of OSF generated in the shape of a ring or OSF exists when thermal oxidation processing is carried out, and is detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration not existing in the whole wafer surface.
- [Claim 2] The silicon single crystal wafer indicated to claim 1 characterized by the oxygen density of said wafer being less than 24 ppmas.
- [Claim 3] The consistency of OSF generated when said thermal oxidation processing is carried out is 2 100 pieces/cm. Silicon single crystal wafer indicated to claim 1 or claim 2 characterized by being the following.
- [Claim 4] [ when manufacturing a silicon single crystal with the Czochralski method ] The silicon single crystal raised sets the temperature gradient between 1400 degrees C to G (the amount of temperature changes / crystal orientation die length) [\*\*/cm] from the melting point near [ under crystal ] the solid-liquid interface at the time of crystal growth. The manufacture approach of the silicon single crystal characterized by \*\*G pulling up as 0 or negative when the difference of the temperature gradient Gc of a crystal center part [\*\*/cm] and the temperature gradient germanium of a crystal circumference part [\*\*/cm] is expressed with \*\*G= (germanium-Gc).
- [Claim 5] [ when manufacturing a silicon single crystal with the Czochralski method ] The silicon single crystal raised sets the temperature gradient between 1400 degrees C to G (the amount of temperature changes / crystal orientation die length) [\*\*/cm] from the melting point near [ under crystal ] the solid-liquid interface at the time of crystal growth. When the difference of the temperature gradient Gc of a crystal center part [\*\*/cm] and the temperature gradient germanium of a crystal circumference part [\*\*/cm] is expressed with \*\*G= (germanium-Gc), In the defective distribution map in which having controlled whenever [ furnace temperature ] so that \*\*G became 0 or negative, and having set the axis of abscissa the axis of ordinate as the crystal diameter as the pull-up rate, and having shown defective distribution The manufacture approach of the silicon single crystal characterized by pulling up a crystal, controlling within the limits of the pull-up rate corresponding to the minimum value of the inside line of an OSF field, and the pull-up rate corresponding to the minimum value of the outside line of an OSF field when an OSF field forms band-like reverse the mold of M characters.
- [Claim 6] The manufacture approach of the silicon single crystal indicated to claim 4 or claim 5 characterized by making precision of the pull-up rate at the time of said crystal growth less than into the average\*\*0.01 of the pull-up rate computed for every growth die length of 10cm of a crystal standard diameter portion [mm/min].
- [Claim 7] The manufacture approach of the silicon single crystal indicated in any 1 term of claim 4 characterized by preparing an annular solid-liquid interface heat insulator in pull-up equipment, and setting spacing on this and the front face of melt as 5-10cm in order to control whenever [ said furnace temperature ] thru/or claim 6.
- [Claim 8] The silicon single crystal wafer characterized by being obtained from the silicon single crystal obtained by the approach of claim 4 thru/or claim 7.

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to a silicon single crystal wafer with few crystal defects, and its manufacture approach.

[0002]

[Description of the Prior Art] In recent years, the quality demand to the silicon single crystal produced with the Czochralski method (it is hereafter written as a CZ process) used as the substrate has been increasing with detailed-izing of the component accompanying high integration of a semiconductor circuit. The defect of a single crystal growth reason in which the oxide film proof-pressure property especially called grown-in (Grown-in) defects, such as FPD, LSTD, and COP, and the property of a device are worsened exists, and importance is attached to reduction of the consistency and size.

[0003] In explaining these defects, it explains being known generally about the factor which determines each concentration of the point defect of the hole mold first called the Vacancy (it may outline Vacancy and Following V) incorporated by the silicon single crystal, and the mold silicon point defect between grids called Interstitial-Si (it may outline Interstitial-Si and Following I) incorporated.

[0004] In a silicon single crystal, V fields are Vacancy, i.e., the crevice generated from lack of a silicon atom, and a field with many things like a hole. With an I region It is the thing of a field with many lumps of the rearrangement and the excessive silicon atom which are generated when a silicon atom exists in an excess. Between V field and an I region The neutral (it may outline Neutral and Following N) field without lack of an atom or an excess (few) will exist. And with [ even if said grown-in defects (FPD, LSTD, COP, etc.) occur when V and I are in a condition / \*\*\*\*\*/ to the last, and it has the bias of some atoms ] saturation [ below ], it has turned out that it does not exist as a defect.

[0005] The concentration of both this point defect is decided from the pull-up rate (growth rate) of the crystal in a CZ process, and relation with the temperature gradient G near [ under crystal ] the solid-liquid interface. The defect called OSF (an oxidation induction stacking fault, Oxidation Induced Stacking Fault) near [ boundary ] V field and an I region When it sees in the cross section of the perpendicular direction to a crystal growth shaft, being distributed in the shape of a ring (it being hereafter called an OSF ring) is checked.

[0006] The defect of these crystal growth reason is acquired as a defective distribution map as shown in drawing 5 , when a crystal orientation changes a growth rate from a high speed to a low speed with CZ pull-up machine with which the temperature gradient G near the solid-liquid interface used the large structure in a furnace (hot zone: it may be called HZ) during the usual crystal.

[0007] And the field where grown-in defects, such as FPD considered as the void reason to which hole type point defects gathered when a growth rate was a high speed comparatively with the above before and after 0.6 mm/min when these were classified according to the direction of the diameter of a crystal (field) as it was shown in drawing 6 for example, LSTD, and COP, exist in high density throughout the direction of the diameter of a crystal, and these defects exist is called the V-rich field (refer to the line (A) of drawing 5 , and drawing 6 (A)). Moreover, when a growth rate is 0.6 or less mm/min, the field where an OSF ring is generated from the circumference of a crystal, the defect of ratios of length to diameter (Large Dislocation: the code of the dislocation loop between grids, LSEPD, LFPD, etc.) considered to be dislocation loop reasons by the outside of this ring exists in a low consistency with lowering of a growth rate, and these defects exist is called the I-rich field (it may be called a ratio-of-length-to-diameter field). Furthermore, if a growth rate is made into a low

speed below 0.4 mm/min order, an OSF ring will condense and disappear at the core of a wafer, and the whole surface will serve as an I-rich field (the line (C) of drawing 5 , drawing 6 (C)).

[0008] Moreover, the existence of the field where neither FPD of a hole reason, LSTD, COP nor LSEPD of a dislocation loop reason and LFPD exist called N field to the outside of an OSF ring is discovered in the medium of a V-rich field and an I-rich field recently. It is reported that this field is the I-rich field side which is not so rich as there is almost no precipitation of oxygen by being in the outside of an OSF ring when oxygen precipitation heat treatment is performed and the contrast of a deposit is checked by X-ray observation etc., and LSEPD and LFPD are formed (refer to the line (B) of drawing 5 , and drawing 6 (B)).

[0009] And improve the temperature distribution in furnace of a pull-up machine for N field which exists only in the pole of a wafer part in the conventional CZ pull-up machine, and a pull-up rate is adjusted. V/G value (when setting a crystal pulling rate to V [mm/min] and setting the average of inclination to G [°/mm] from the melting point of silicon whenever [ crystal internal temperature / of the pull-up shaft orientations between 1300 degrees C ]) If it controls to the whole wafer surface and a crystal overall length by setting to 0.20-0.22mm<sup>2</sup> / °, and min the ratio expressed with V/G, and it is possible to extend N field all over a wafer, it has proposed (JP,8-330316,A).

[0010] However, if it is going to extend and manufacture such a super-low defective field into the whole crystal, since this field will be limited only to N field by the side of an I-rich field, if it is an experimental aircraft, it is [ with a production machine, / precision control is difficult and / a difficulty ] in productivity at any rate, and is not practical [ a control range is very narrow on manufacture conditions, and ].

[0011] When the present usual silicon single crystal, on the other hand, performs operation which changes a growth rate from a high speed to a low speed intentionally in a crystal orientation as shown in drawing 5 R> 5, As shown in drawing 6 , a whole (A) surface V-rich field mold, the coexistence mold of a (B) V-rich field and N-field, (C) A whole surface I-rich field mold (it may be called a ratio-of-length-to-diameter rich field mold), and a (D) V-rich field and an I-rich field coexistence mold (un-illustrating) are formed, and the growth rate of a crystal orientation is adjusted and manufactured so that each quality may be acquired according to the object application.

[0012] And the whole surface V-rich field mold of (A) is mass-produced as a reference standard among these. At a device process, although the V-N coexistence mold of (B) is manufactured as an amelioration article of (A), even if N-field is a high yield, it falls in a V-rich field and is imperfect. Although the whole surface I-rich field mold of (C) is manufactured as a particle monitor, ratio of length to diameter serves as a failure, and is not used as an object for device production. Moreover, even if it throws (A), (C), and the wafer of (D) each type into a device process, they have the inclination for the yield of a device to get worse, under the effect of a hole, the rearrangement between grids, etc. with the large size which remains on the wafer front face.

[0013] It is [ a difficulty ] in productivity and is not practical as it furthermore mentioned above recently, although the whole surface N-field mold was proposed as a (E) type (un-illustrating). Moreover, although the single crystal with which an OSF ring is generated, the nucleus of OSF exists, and FPD and ratio of length to diameter do not exist in the whole surface is proposed as a (F) type when thermal oxidation processing is carried out in a whole surface N-field (Japanese Patent Application No. No. 325428 [ nine to ]), the Vacancy defect still more detailed than FPD may exist, and such a defect is detected by for example, Cu decoration. And it is the cause by which this degrades oxide-film pressure-proofing, and an improvement was desired further.

[0014]

[Problem(s) to be Solved by the Invention] This invention aims at obtaining the silicon single crystal wafer by the CZ process to which the defect detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration does not exist in the whole wafer surface under the stable manufacture condition, although the potential nucleus of OSF or OSF generated in the shape of a ring exists by the super-low consistency when it is made in view of such a trouble and thermal oxidation processing is carried out.

[0015]

[Means for Solving the Problem] Invention which it was accomplished in order that this invention might attain said object, and was indicated to claim 1 of this invention is the silicon single crystal wafer raised by the Czochralski method, and is a silicon single crystal wafer characterized by for the defect which the nucleus of OSF generated in the shape of a ring or OSF exists when thermal oxidation processing is carried out, and is detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration not to exist in the whole wafer

surface. Thus, although an OSF ring or its potential nucleus exists in the wafer of this invention, there are no grown-in defects, such as FPD, and the defect detected especially with Cu decoration does not exist.

[0016] And as indicated to claim 2 in this case, it is desirable that the oxygen densities of a wafer are under 24ppma(s) (ASTM'79 value). Although the nucleus of OSF exists when thermal oxidation processing is carried out if it does in this way, OSF can obtain the silicon single crystal wafer with which the defect which does not occur and is detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration does not exist in the whole wafer surface.

[0017] For invention furthermore indicated to claim 3, the consistency of OSF generated when said thermal oxidation processing is carried out is 2 100 pieces/cm. It is the silicon single crystal wafer of the super-low defect in which it is the following. In this case, measurement of an OSF consistency measured by carrying out etching processing with light (Wright) liquid to it, after performing heat treatment for 1200 degrees C / 100 minutes to the silicon single crystal wafer.

[0018] and as the manufacture approach of such a silicon single crystal wafer [ as indicated to claim 4 of this invention, when manufacturing a silicon single crystal with the Czochralski method ] The silicon single crystal raised sets the temperature gradient between 1400 degrees C to G (the amount of temperature changes / crystal orientation die length) [ $**/\text{cm}$ ] from the melting point near [ under crystal ] the solid-liquid interface at the time of crystal growth. When the difference of the temperature gradient  $G_c$  of a crystal center part [ $**/\text{cm}$ ] and the temperature gradient germanium of a crystal circumference part [ $**/\text{cm}$ ] is expressed with  $**G = (\text{germanium} - G_c)$ , it is the manufacture approach of the silicon single crystal characterized by  $**G$  pulling up as 0 or negative.

[0019] [ when invention furthermore indicated to claim 5 of this invention manufactures a silicon single crystal with the Czochralski method ] The silicon single crystal raised sets the temperature gradient between 1400 degrees C to G (the amount of temperature changes / crystal orientation die length) [ $**/\text{cm}$ ] from the melting point near [ under crystal ] the solid-liquid interface at the time of crystal growth. When the difference of the temperature gradient  $G_c$  of a crystal center part [ $**/\text{cm}$ ] and the temperature gradient germanium of a crystal circumference part [ $**/\text{cm}$ ] is expressed with  $**G = (\text{germanium} - G_c)$ , In the defective distribution map in which having controlled whenever [ furnace temperature ] so that  $**G$  became 0 or negative, and having set the axis of abscissa the axis of ordinate as the crystal diameter as the pull-up rate, and having shown defective distribution When an OSF field forms band-like reverse the mold of M characters, it is the manufacture approach of the silicon single crystal characterized by pulling up a crystal, controlling within the limits of the pull-up rate corresponding to the minimum value of the inside line of an OSF field, and the pull-up rate corresponding to the minimum value of the outside line of an OSF field. Here, an OSF field shows distribution of the OSF ring of crystal growth shaft orientations.

[0020] The defective distribution map of drawing 1 which analyzed and searched for the result of an experiment and examination thus, to origin If a crystal is pulled up controlling [ control whenever / furnace temperature / so that difference  $**G$  of the crystal center of Inclination G and the crystal circumference becomes 0 or negative from the melting point of silicon whenever / crystal internal temperature / of the pull-up shaft orientations between 1400 degrees C /, and ] within limits which specified the pull-up rate above The defect which the nucleus of OSF generated in the shape of a ring or OSF exists when thermal oxidation processing indicated to claim 1 is carried out, and is detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration can produce the silicon single crystal which does not exist in the whole wafer surface.

[0021] And as indicated to claim 6 in this case, it is desirable to make precision of the pull-up rate at the time of said crystal growth less than into the average  $**0.01$  [mm/min] of the pull-up rate computed for every growth die length of 10cm of a crystal standard diameter portion (the body part of a single crystal is said). If precision of a pull-up rate is made into high degree of accuracy in this way, under the conditions easily specified by claim 4, it is stabilized and a silicon single crystal can be manufactured.

[0022] Moreover, what is necessary is to prepare an annular solid-liquid interface heat insulator in pull-up equipment, and just to set spacing on this soffit and the front face of melt as 5-10cm, in order to control whenever [ furnace temperature ], as indicated to claim 7. If it carries out like this, the temperature gradient of the 0 or negative, i.e., crystal, circumference and the temperature gradient of a crystal center have equal difference  $**G = (\text{germanium} - G_c)$  of the temperature gradient  $G_c$  of the above-mentioned crystal center part [ $**/\text{cm}$ ], and the temperature gradient germanium of a crystal circumference part [ $**/\text{cm}$ ], or whenever

[ furnace temperature ] can be controlled so that the direction of the temperature gradient of the crystal circumference becomes lower than a crystal center, and defective distribution will serve as reverse a mold of M characters.

[0023] And the silicon single crystal wafer (claim 8) which slices the silicon single crystal manufactured by the manufacture approach of above-mentioned claim 4 thru/or a silicon single crystal according to claim 7, and is obtained turns into a silicon single crystal wafer with which the defect detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration does not exist in the whole wafer surface, although the nucleus of OSF generated in the shape of a ring or OSF exists like claim 1 when thermal oxidation processing is carried out to a wafer.

[0024] Hereafter, although explained to a detail per this invention, this invention is not limited to these. In advance of explanation, lessons is taken from each vocabulary, and it explains beforehand.

1) K2 Cr 2O7 after cutting down a wafer from the silicon single crystal rod after growth and etching and removing a surface distortion layer with the mixed liquor of fluoric acid and a nitric acid in FPD (Flow Pattern Defect) A pit and a ripple pattern arise by etching a front face with the mixed liquor of fluoric acid and water (Secco etching). This ripple pattern is called FPD, and the defects of oxide-film pressure-proofing increase in number, so that the FPD consistency within a wafer side is high (refer to JP,4-192345,A).

[0025] 2) When the same Secco etching as FPD is performed, call SEPD (Secco Etch Pit Defect) a thing without FPD, a call, and a flow pattern for the thing accompanied by a flow pattern (flow pattern) with SEPD. When it is thought in this that large SEPD (LSEPD) 10 micrometers or more originates in a rearrangement cluster and a rearrangement cluster exists in a device, a current leaks through this rearrangement and it stops achieving the function as a P-N junction.

[0026] 3) Cut down a wafer from the silicon single crystal rod after growth, and carry out cleavage of the wafer to LSTD (Laser Scattering Tomography Defect) after etching and removing a surface distortion layer with the mixed liquor of fluoric acid and a nitric acid. Incidence of the infrared light can be carried out from this cleavage plane, and the scattered light by the defect which exists in a wafer can be detected by detecting the light which came out from the wafer front face. About the scatterer observed here, it is an institute etc., there is already a report, and it is regarded as the oxygen sludge (Jpn.J.Appl.Phys. Vol.32, P3679, 1993 reference). Moreover, the result that it is the void (hole) of octahedron is also reported by the latest research.

[0027] 4) the defect which becomes the cause of degrading oxide film pressure-proofing of the core of a wafer, with COP (Crystal Originated Particle) -- it is -- Secco -- by SC-1 washing (washing by the mixed liquor of NH4 OH:H2 O2:H2 O=1:1:10), the defect set to FPD if dirty works as a selection etching reagent, and is set to COP. The diameter of this pit is investigated with light scattering measurement by 1 micrometer or less.

[0028] 5) It is the defect which there are LSEPD, LFPD, etc. in ratio of length to diameter (Large Dislocation: code of the dislocation loop between grids), and is considered to be a dislocation loop reason. A large thing 10 micrometers or more is said that LSEPD described above also in SEPD. Moreover, also in FPD which LFPD described above, the magnitude of a head pit says a large thing 10 micrometers or more, and it is considered the dislocation loop reason also here.

[0029] 6) Cu decoration is the approach of observing a crystal defect with an infrared microscope by carrying out thermal diffusion of the impurity (Cu) intentionally, quenching after saturation, and depositing an impurity to a defect, and it is effective in detection of a Vacancy defect (V defect: hole mold point defect) still more detailed than FPD etc.

[0030] the place investigated in the detail about the boundary neighborhood of V field and an I region about the silicon single crystal growth by the CZ process as this invention persons proposed by Japanese Patent Application No. No. 199415 [ nine to ] previously -- \*\*\*\* of this boundary neighborhood -- the narrow field had few FPD(s), LSTD(s), and COP remarkably, and it discovered that there was a neutral field where ratio of length to diameter does not exist, either.

[0031] Then, if this neutral field can be extended all over a wafer, it will conceive that a point defect can be reduced substantially, and since the pull-up rate is almost fixed in the wafer side of a crystal, the main factors which determine concentration distribution of the point defect within a field will be temperature gradients in a growth (pull-up) rate and the relation of a temperature gradient. That a difference is in the temperature gradient of shaft orientations in a wafer side that is, on a problem If this difference can be reduced, that the concentration difference of the point defect within a wafer side can also be reduced A header, When controlling whenever

[ furnace temperature ] and adjusting the pull-up rate so that the difference of the temperature gradient  $G_c$  of the crystal center section and the temperature gradient germanium of a crystal circumference part might be set to  $**G = (\text{germanium} - G_c) \leq 5 \text{ degree-C/cm}$ , a wafer without the defect which the whole wafer surface becomes from N field came to be obtained.

[0032] In this invention, as a result of having used the crystal pulling equipment by the small CZ process, difference  $**G$  of the above temperature gradients rearranging the structure in a furnace, changing  $**G$ , changing a pull-up rate and investigating the inside of the crystal face, the following knowledge was newly acquired. Spacing from the surface of hot water to the soffit of an annular solid-liquid interface heat insulator is changed,  $**G$  was changed, to 0.6 - 0.3 mm/min, it slows down 0.05 mm/min of average pull-up rates at a time, they were changed every 10cm, the OSF ring disappeared focusing on crystal bulk, and the used structure in a furnace investigated signs that a ratio-of-length-to-diameter field was formed, as shown in drawing 4 (a).

[0033] The result was analyzed and it was shown in drawing 1 as a defective distribution map. An axis of abscissa is the crystal diameter direction, and an axis of ordinate is a growth rate. When the temperature gradient germanium of the 0 or negative, i.e., crystal, circumference and the temperature gradient  $G_c$  of a crystal center have equal  $**G$  or the temperature gradient germanium of drawing 1 of the crystal circumference is lower than the temperature gradient  $G_c$  of a crystal center, in the range of 0.50 - 0.48 mm/min, OSF of a growth rate is beltlike, and it means being distributed over the reverse mold of M characters. And although an OSF ring or its potential nucleus exists like drawing 3 if a growth rate looks at the range of 0.50 - 0.48 mm/min as the crystal face among this distribution, it turns out that parts other than this serve as a wafer which is N-field.

[0034] The greatest description of this wafer is that N-field of parts other than an OSF field is all an N-field by the side of I-rich. That is, although the inside of an OSF ring should originally become the V-rich side (refer to the line (B) of drawing 5, and drawing 6 (B)), in this invention, the inside of an OSF ring and an outside serve as N-field by the side of I-rich. Therefore, the defect which should be detected with Cu decoration besides FPD and a COP mold is also eliminated from the whole wafer surface.

[0035] And the pull-up rate corresponding to the minimum value of the inside line of an OSF field when, as for this invention, an OSF field forms band-like reverse the mold of M characters in the defective distribution map of drawing 1, It is a thing called the method of a pull-up in a crystal, controlling a pull-up rate within the limits of the pull-up rate corresponding to the minimum value of the outside line of an OSF field. If it says concretely in the above-mentioned example, a growth rate will be set up within the limits of 0.50 - 0.48 mm/min, and it will control and pull up to high degree of accuracy so that it may become less than  $**0.01 \text{ mm/min}$  of averages of the target growth rate computed for every growth die length of 10cm of a crystal standard diameter portion. In this way, the vertical division of the obtained single crystal rod was carried out, and defective distribution was investigated like the above. The result is shown in drawing 2. The overall length is covered, an OSF field is distributed in the shape of a ring, and, as for the part which maintained and pulled up the optimal growth rate so that clearly from drawing 2, it turns out that the whole surface other than an OSF field is N-fields.

[0036]  $**G$  exceeds 0. Conversely, in plus (i.e., when the temperature gradient germanium of the crystal circumference is higher than the temperature gradient  $G_c$  of a crystal center) As the growth rate showed the range of about 0.6 to 0.4 mm/min to drawing 5, OSF is beltlike and it is distributed over the U character mold. It turns out that the wafer with which it is formed only in N-field by the side of [ like this invention article ] whole surface I-rich, and an OSF ring or its potential nucleus exists does not appear even if it sees with the crystal-face interior division cloth of drawing 6. The above thing is not discovered when the conventional  $**G$  experiments on a plus side with large crystal pulling equipment, but as a result of investigating the crystal with which the  $**G$  above-mentioned this time used 0 or negative crystal pulling equipment, it is discovered.

[0037] About whenever [ furnace temperature / of the pull-up equipment in this examination ], as a result of analyzing wholeheartedly using the comprehensive heat transfer analysis software FEMAG (F. 33 Dupret, P.Nicodeme, Y.Ryckmans, P.Wouters, and M.J.Crochet, Int.J.HeatMass Transfer, 1849 (1990)), it becomes clear.

[0038] It turns out that OSF is not generated by thermal oxidation processing and a device is not affected on the other hand about OSF which exists in the wafer of this invention even if the nucleus of OSF exists when it considers as hypoxia concentration in the whole wafer surface from the latest research. It was checked [ as a result of using the same crystal pulling equipment for the threshold value of this oxygen density and pulling up



the crystal of some kinds of oxygen density level, ] that OSF does not occur when the oxygen densities in the whole wafer surface were under 24ppma(s) (ASTM'79), and thermal oxidation processing of a wafer was performed.

[0039] That is, although it is to 24ppma(s) that according to examination the nucleus which serves as OSF covering a crystal overall length exists when an oxygen density is gradually lowered while raising the crystal of one, but OSF is observed when thermal oxidation processing of a wafer is performed and the nucleus of OSF existed in less than 24 ppmas, it turned out that OSF by thermal oxidation processing is not generated.

[0040] Incidentally, in order to set the oxygen density under growth crystal to less than 24 ppmas, that what is necessary is just to carry out by the approach generally used from the former, distribution etc. can be adjusted whenever [ rotational frequency / of a crucible /, or melt internal temperature ], and the means of controlling the convection current of melt can perform easily.

[0041] In addition, even if an OSF ring is not generated, there is an inclination for precipitation of oxygen to decrease, in the place where the nucleus exists, but since strong gettering is not required in low-temperature-izing of a device process in recent years, either, the little of the precipitation of oxygen in an OSF ring does not become a problem.

[0042]

[Embodiment of the Invention] Hereafter, the gestalt of operation of this invention is explained to a detail, referring to a drawing. First, drawing 4 R> 4 (a) explains the example of a configuration of the crystal pulling equipment by the CZ process used by this invention. As shown in drawing 4 (a), this crystal pulling equipment 30 The pull-up room 31, the crucible 32 prepared all over the pull-up room 31, and the heater 34 arranged around a crucible 32, It has the reel style (not shown) which rotates or rolls round the crucible maintenance shaft 33 made to rotate a crucible 32 and its rolling mechanism (not shown), the seed chuck 6 holding the seed crystal 5 of silicon, the wire 7 that pulls up a seed chuck 6, and a wire 7, and is constituted. A quartz crucible is prepared in the side in which a crucible 32 holds the silicon melt (molten bath) 2 of the inside, and the graphite crucible is prepared in the outside. Moreover, the heat insulator 35 is arranged around [ outside ] the heater 34. [0043] Moreover, in order to set up the manufacture conditions in connection with the manufacture approach of this invention, the annular solid-liquid interface heat insulator 8 is formed in the periphery of the solid-liquid interface 4 of a crystal. This solid-liquid interface heat insulator 8 forms the spacing 10 of 5-10cm between that soffit and surface of hot water 3 of silicon melt 2, and is installed in it. The up heat insulator 9 prepared on the heat insulator 35 which surrounds the heater shown in drawing 4 (b) will be used according to conditions whenever [ furnace temperature ], and spacing 10 will be adjusted in that case. Furthermore, coolant gas may be sprayed or the tubed cooling system (un-illustrating) which interrupts radiant heat and cools a single crystal may be formed. Independently, by installing the magnet which is not illustrated in the horizontal outside of the pull-up room 31, and impressing magnetic fields, such as a horizontal direction or a perpendicular direction, to silicon melt 2, the convection current of melt is controlled and, recently, the so-called MCZ method for measuring the stable growth of a single crystal is used in many cases.

[0044] Next, the single-crystal-growth approach by above crystal pulling equipment 30 is explained. First, within a crucible 32, the high grade polycrystal raw material of silicon is heated more than the melting point (about 1420-degreeC), and is dissolved. Next, the head of seed crystal 5 is made contacted or immersed in the surface abbreviation core of melt 2 by beginning to roll a wire 7. Then, while rotating the crucible maintenance shaft 33 in the proper direction, single crystal growth is started by rolling round rotating a wire 7 and pulling up seed crystal 5. Henceforth, the single crystal rod 1 of an approximate circle column configuration can be obtained by adjusting a pull-up rate and temperature appropriately.

[0045] In this invention, that in this case, it is important especially in order to attain the object of this invention As shown in drawing 4 (a) or drawing 4 R> 4 (b), it sets to the periphery space of the liquefied part in the single crystal rod 1 on the surface of hot water of the pull-up room 31. It is having formed the annular solid-liquid interface heat insulator 8 so that the temperature region from the melting point of the crystal near the surface of hot water to 1400 degrees C could be controlled, and having arranged the up heat insulator 9 on a heat insulator 35.

[0046] Namely, what is necessary is to form the annular solid-liquid interface heat insulator 8 in the pull-up room 31, and just to set the spacing 10 on this soffit and the front face of melt as 5-10cm, as shown in drawing 4 (a) in order to control whenever [ this furnace temperature ]. If it carries out like this, the temperature gradient



of the 0 or negative, i.e., crystal, circumference and the temperature gradient of a crystal center have equal difference  $G = (G_{\text{germanium}} - G_c)$  of the temperature gradient  $G_c$  of the above-mentioned crystal center part [ $^{\circ}\text{C}/\text{cm}$ ], and the temperature gradient germanium of a crystal circumference part [ $^{\circ}\text{C}/\text{cm}$ ], or whenever [furnace temperature] can be controlled so that the direction of the temperature gradient of the crystal circumference becomes lower than a crystal center. As shown in drawing 4 (b), while adjusting the above-mentioned spacing 10 as an option, there is also the approach of adding the up heat-insulating element 9 on a heat insulator 35, and controlling the heat dissipation from up space.

[0047] moreover, about the precision of the pull-up rate at the time of said crystal growth If it is desirable to carry out to less than the average  $\pm 0.01$  of the pull-up rate computed for every growth die length of 10cm of a crystal standard diameter portion [ $\text{mm}/\text{min}$ ] and the precision of a pull-up rate is in this range Although a crystal overall length is covered and there is the potential nucleus in an OSF field according to the synergistic effect of whenever [above-mentioned furnace temperature], ( $G$ ) and a pull-up rate condition value, the other whole surface within a field can stabilize for it and manufacture the silicon single crystal which is N-field.

[0048] The silicon single crystal wafer which slices the silicon single crystal manufactured by the manufacture approach of the silicon single crystal described above, and is obtained When thermal oxidation processing is carried out to a wafer, the nucleus of OSF generated in the shape of a ring or OSF exists. And the consistency of OSF generated when the defect detected with FPD, COP, ratio of length to diameter, LSTD, and Cu decoration does not exist in the whole wafer surface but carries out thermal oxidation processing is the super-low defective article of two or less [ $100 // \text{cm}$ ].

[0049]

[Example] Hereafter, although the example of this invention is given and explained, this invention is not limited to these.

(Example 1) With the pull-up equipment 30 shown in drawing 4 (a), 100kg of raw material polycrystalline silicon was charged to the 24 inch quartz crucible, and the silicon single crystal rod with the diameter of 8 inches, a bearing  $\langle 100 \rangle$ , and a body die length of about 1m was pulled up. The used structure in a furnace (hot zone: HZ) set the spacing 10 of the surface of hot water 3 and the soffit of the annular solid-liquid interface heat insulator 8 as 70mm, and held the water temperature of silicon melt 2 at about 1420 degrees C.

[0050] Under the above conditions, it slows down 0.05 mm/min of average pull-up rates at a time, they were changed every 10cm, to 0.6 - 0.3 mm/min, the OSF ring disappeared focusing on crystal bulk, and signs that a ratio-of-length-to-diameter field was formed were investigated. The search procedure carried out the vertical division of the crystal to 2mm in thickness, carried out etching clearance of the processing distortion of a front face, and produced the sample of two sheets. One sheet observed FPD and ratio of length to diameter, after performing SEKO etching during 30 minutes. Moreover, about the one remaining sheets, after performing heat treatment for 1200 degrees C / 100 minutes, etching processing was carried out with the Wright reagent, and the generating situation of OSF was checked. The result was collectively shown in drawing 1 as a defective distribution map. An axis of abscissa is the crystal diameter direction, and an axis of ordinate is a pull-up rate. Drawing to OSF is beltlike and it turns out that it is distributed over the reverse mold of M characters. When this is seen, in order to obtain this invention article, with the structure in this furnace, it turns out that what is necessary is just to control a growth rate to 0.48 - 0.50 mm/min.

[0051] Next, although an OSF ring exists in the crystal face, in order to expand this invention article whose whole surface other than an OSF field is N-fields to a crystal orientation based on the above-mentioned examination and experimental result, it was set as the optimal growth rate (0.5 - 0.48 mm/min), and it controlled and pulled up so that it might become the less than  $\pm 0.01$  averages of the target growth rate computed for every growth die length of 10cm of a crystal standard diameter portion. In this way, the vertical division of the obtained single crystal rod was carried out, and  $\pm 0.01$  was investigated like the above. The result is shown in drawing 2. The overall length is covered, an OSF field is distributed in the shape of a ring, and, as for the part which maintained and pulled up the optimal growth rate so that clearly from drawing, it turns out that the whole surface other than an OSF field is N-fields.

[0052] The single crystal rod was independently processed into the pull-up and the mirror-polishing finishing wafer on the above and these conditions, and FPD, ratio of length to diameter, OSF, LSTD, etc. were evaluated. Consequently, although the OSF ring existed in the center section as shown in drawing 3, the whole surface within a field other than an OSF field was the wafers which are N-fields. About 50 consistencies /of OSF in an

OSF field were [ cm ] 2, and were low consistencies. When Cu decoration was furthermore given, the hole mold defect was not generated in N-field section. In addition, the oxide-film proof-pressure property of this wafer became 100% of rates of C-mode excellent article. The C-mode Measuring condition is as follows.

1) Oxide-film thickness : 25nm Two measuring electrode: Phosphorus dope polish recon and 3 electrode-surface product:8mm2 4 judging current: 2 and 5 excellent-article judging:dielectric-breakdown electric field judged 1mA /of things of 8 or more MV/cm cm to be an excellent article.

[0053] (Example 2) As shown in drawing 4 (b), the up heat insulator 9 was installed for the structure in a furnace on the heat insulator 35, and except having set spacing 10 of the silicon melt side 3 and the soffit of the annular solid-liquid interface heat insulator 8 to 60mm, as a result of pulling up on the same conditions as an example 1, the single crystal rod of the almost same quality as an example 1 was obtained. In this case, as a result of \*\*G's shifting to a minus side further, the inclination for the width of face of the OSF field within a wafer side to become narrow was seen.

[0054] (Example 3) OSF was not generated even if it carried out oxidation treatment except having held down the oxygen density under growth crystal to 24 or less ppmas, when an example 1 and these conditions estimated the pull-up and the defect. That is, it is thought that it is what is not generated even if it carries out thermal oxidation processing since this is hypoxia although it is thought that the potential nucleus of OSF exists.

[0055] In addition, this invention is not limited to the above-mentioned operation gestalt. The above-mentioned operation gestalt is instantiation, and no matter it may be what thing which has the same configuration substantially with the technical thought indicated by the claim of this invention, and does the same operation effectiveness so, it is included by the technical range of this invention.

[0056] For example, in the above-mentioned operation gestalt, although the example was given and explained per when a silicon single crystal with a diameter of 8 inches was raised, this invention is not limited to this but can be applied also to the diameter of 10-16 inches, or the silicon single crystal beyond it. Moreover, it cannot be overemphasized that this invention is applicable also to the so-called MCZ method for impressing a level magnetic field and length magnetic field, a cusp field, etc. to silicon melt.

[0057]

[Effect of the Invention] As explained above, according to this invention, the wafer to which the maximum N field where the nucleus of OSF generated in the shape of a ring or OSF exists in when thermal oxidation processing is carried out, and FPD, COP, ratio of length to diameter, LSTD, and the defect detected especially with Cu decoration do not exist in the whole wafer surface was expanded is easily producible. And if hypoxia-ization is used together, OSF is not generated, either but the whole parenchyma top wafer surface can manufacture a defect-free silicon single crystal wafer.

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[Translation done.]

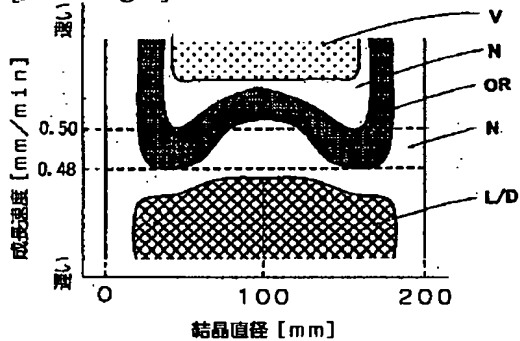
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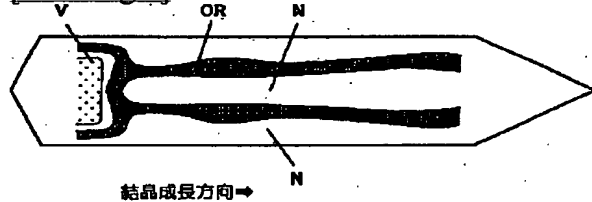
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## DRAWINGS

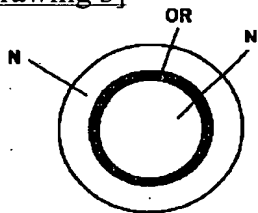
[Drawing 1]



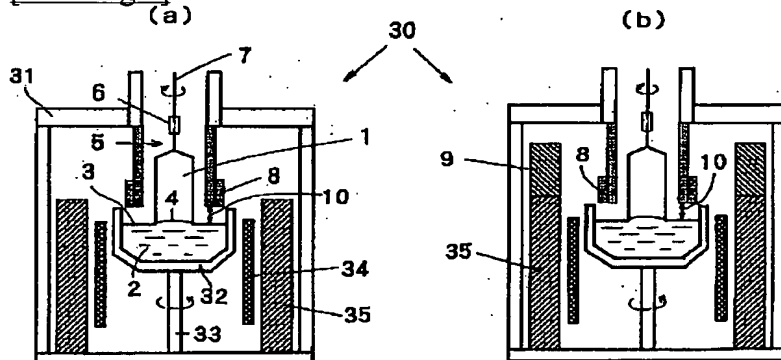
[Drawing 2]



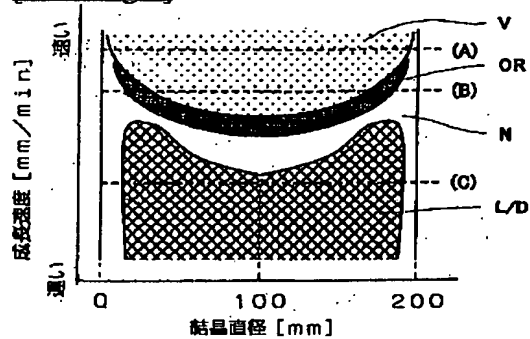
[Drawing 3]



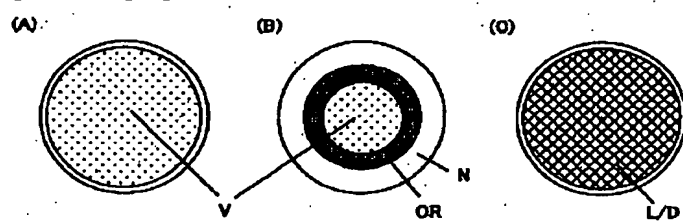
[Drawing 4]



[Drawing 5]



[Drawing 6]



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